

2. SURVEY METHODOLOGY

The field sampling methods used to collect the required data are presented in this section. The logic used for selecting the appropriate number of samples was based on the allowed decision error, and the metals chosen for analysis were based on historical use of metals (see Table 1.1). These topics are discussed first followed by a description of each survey activity. Survey activities are described in terms of the sampling methods, problems encountered, discrepancies noted, quality assurance applied, and information documented. The major survey activities conducted include:

- metal wipe samples,
- PCB wipe samples,
- bulk dust for metals analysis,
- roof samples for PCBs and metals analysis,
- paint for metals analysis,
- perchlorate risk survey,
- physical hazards assessment, and
- historical data overview.

Limits on Decision Error. Two different confidence levels were established for identifying contaminated areas in the building. One confidence level was used to prove anticipated clean areas were indeed free from contamination, and the other confidence level was used to prove the existence of contamination in areas where contamination was expected. A conservative confidence level of 95% was selected for verifying clean areas, and a more liberal confidence level of 65% was selected for verifying contaminated areas. A conservative confidence level was chosen for verifying clean areas due to the higher risk to workers should the clean assumption be false [i.e., appropriate personal protective equipment (PPE) not worn]. On the other hand, a more liberal confidence level was chosen for the areas assumed to be contaminated because the appropriate PPE and other precautions would be taken.

The number of samples that must be collected to meet a certain confidence level will depend on the number of sampling points available and the allowed bound on the error as expressed in the following equation:

$$n = \frac{Npq}{[(N-1)D] + pq}$$

where:

- n = required number of samples,
- N = population (number of sampling points available),
- p = population portion below action limit (confidence level),
- q = 1 - p,
- D = B² / 4,
- B = bound on error estimation.

This approach assumes that the area to be sampled is relatively homogeneous. Therefore, the building was divided into broad areas based on anticipated contamination (i.e., office areas, beryllium processing areas, radiological areas, and process areas), and then these broad areas were further divided into HAs as described in Sect. 1. The number of sampling points available was then based on the number of locations that could be contaminated through visual observation of a particular HA within a building area.

When the described approach is applied, more samples are required from assumed clean areas than from areas assumed to be contaminated. For example, if 100 potentially contaminated locations are identified in an HA, 45 samples would be needed to verify that the area is clean, but only 7 samples would be needed to verify that the area is contaminated. If any sample from a particular HA within a building area was found to be contaminated, the entire HA within that building area was assumed to be possibly contaminated.

Metals Chosen for Analysis. To provide quality results while minimizing costs, it was decided to analyze the samples for the contaminants of highest concern that were most likely to be present. As a result of this strategy, three analysis suites were established: standard suite, expanded suite, and full suite. The standard suite included the elements Be, Li, Ni, Ti, and U based on their use and LOC. The expanded suite included all elements in the standard suite plus lead and chromium. The expanded suite was adapted to the paint samples because of the higher potential of lead and chromium in these samples. The full suite included common ICP metals and was applied to the ventilation systems and certain other samples to verify that there were not any unknown contaminants that were not identified during the historical review of the building. In addition, thorium was analyzed for a few of the samples to determine whether this element is a concern.

Interference problems were experienced with a number of samples during their analysis. To overcome the interference problems, these samples were reanalyzed using inductively coupled plasma-mass spectroscopy (ICP-MS). Due to these interference problems, the full suite of metals were

analyzed. Chemical analyses for all samples, except thorium, and samples with major interference problems were conducted at Radian's American Industrial Hygiene Association and U.S. Environmental Protection Agency (EPA) accredited laboratory in Austin, Texas . Thorium analyses were conducted by TMA Eberline Thermo Analytical in Albuquerque, New Mexico, and samples with major interferences were analyzed by the University of Hawaii in Honolulu, Hawaii. More discussion on interference problems is provided in Sect. 2.1.

2.1 METAL WIPE SAMPLES

The metal wipe sampling activity was by far the largest sampling activity. Table 2.1 indicates the general locations where wipe samples were gathered. The number of samples and their locations were estimated in the S&T Plan but adjusted during field sampling activities.

Table 2.1. Number and locations for metal wipe samples

HAs	Beryllium areas	Controlled areas	Misc. laboratory areas	Office areas	Total
Exhaust ventilation system	4 (2)	2	6 (1)	NA	12 (3)
Supply ventilation system	9 (1)	7 (1)	22 (4)	8 (2)	46 (8)
Walls	9 (1)	2	5 (1)	9	25 (2)
Rotating equipment	5 (1)	NA	5	NA	10 (1)
Misc. horiz. surfaces >6 ft	7 (1)	7 (2)	12	17 (1)	43 (4)
Misc. horiz. surfaces <6 ft	0	1	5	2 (1)	8 (1)
Misc. equipment	0	4 (2)	15	NA	19 (2)
Total	34 (6)	23 (5)	70 (6)	36 (4)	163 (21)

NA = not available

() = number of QC samples

No major discrepancies were noted during field sampling activities. Some discrepancies in the form of negative concentrations or concentrations greater than 100% were encountered during the ICP analysis of some of the samples. The ICP analyses were conducted at Radian's laboratory using EPA SW-846 Method 6010. Upon further investigation it was noted that these discrepancies were caused by interferences from unknown metals. To determine which elements were causing the interferences and to determine more accurate concentrations, samples with noted discrepancies were re-analyzed using ICP-MS. The ICP-MS analyses were conducted by the University of Hawaii on samples digested by Radian. Samples with noted interference problems and those analyzed using ICP-MS are summarized in Table 2.2. Thorium cannot be analyzed through standard ICP analysis. Therefore, some samples were split and sent to Eberline Analytical Services for thorium analysis.

All sampling was conducted in accordance with the S&T Plan except where field situations required small modifications. General modifications included:

- using more than one filter to ensure all transferable dust was absorbed onto the wipe,
- using a special tool to collect wipes from certain hard to reach locations, and
- not obtaining tare and sample weights for the wipes.

While sampling, it was observed that some dust was not absorbed onto the wipe when wiping areas with heavy layers of dust. It was decided at that time that more than one wipe should be used to ensure the removal of all transferable dust. The sampler used visual observation to ensure all dust was removed from the surface.

Some areas could not be reached by hand to obtain a wipe due to the height of the object or the limited access opening (e.g., vent pipe). To obtain wipes from these areas, a clip attached to an 8-ft handle was used to reach these areas. A clean plastic bag was placed between the clip and the wipe to prevent cross contamination between the handle and sample.

The S&T Plan suggested that tare and sample weights be obtained of the wipes to estimate the gross quantity of dust picked up by the wipe. By knowing the gross quantity of dust on a wipe and the quantity of contaminant on a wipe, the contaminant concentration within the dust may be calculated. It was planned to use scales already available in TA-3-141 for this measurement. Unfortunately, it was found during the field effort that none of the available scales were sufficiently sensitive to measure this small weight difference. Therefore, this information was not obtained.

Table 2.3 presents the results from quality control (QC) samples that were taken during the December 1995 survey. QC samples were obtained by wiping a surface area adjacent to the original sample location. The relative difference was calculated for sample results found above the detection limit. As a result, the relative difference was determined for 46 different analytes from the 20 samples. The relative difference ranged from 0 to 155%, with an average of 32%. Ten of the results demonstrated a relative difference over 50%. The reason for some of the larger relative differences is attributed to the differences in wipe samples when they are taken adjacent to each other. A variation in metal concentrations in dust in two areas would be expected, even if the areas were adjacent to each other.

In addition to the duplicate QC samples given in Table 2.3, two samples spiked with beryllium were obtained from LANL ESH-5 and were also submitted to the Radian laboratory. These samples were spiked with 70 and 100 μg each, and the analytical results were 72.6 and 104 μg , respectively. Both of these results are within the allowed error level.

Table 2.2. Samples with major interference problems

Sample ID and type	Sample location	Building area	HA
1043J - Wipe	Inside FE-6 room 148	General Laboratory Area	Exhaust Ventilation System
1044K - Wipe	Inside FE-6 outdoors	Outdoors	Exhaust Ventilation System
1048R - Bulk	Inside FE-6	General Laboratory Area	Exhaust Ventilation System
1050W - Wipe	Inside FE-6 room 148	General Laboratory Area	Exhaust Ventilation System
1069X - Wipe	Inside FE-9	General Laboratory Area	Exhaust Ventilation System
1070F - Wipe	Inside FE-9	General Laboratory Area	Exhaust Ventilation System
1141X - Bulk	Inside FE-9 south duct	General Laboratory Area	Exhaust Ventilation System
1142Y - Bulk	Inside FE-9 north duct	General Laboratory Area	Exhaust Ventilation System
1144C - Bulk	Inside FE-9 north duct	General Laboratory Area	Exhaust Ventilation System
1053A - Wipe	Inside FE-10 room 136	General Laboratory Area	Exhaust Ventilation System
1066X - Wipe	Inside FE-10	General Laboratory Area	Exhaust Ventilation System
1067Y - Wipe	Inside FE-10	General Laboratory Area	Exhaust Ventilation System
1071G - Wipe	Inside FE-10	General Laboratory Area	Exhaust Ventilation System
1147H - Bulk	Inside FE-10	General Laboratory Area	Exhaust Ventilation System
1032N - Wipe	Top of furnace room 144	General Laboratory Area	Misc. Horizontal Surface Area
1033R - Wipe	Top of firehose room 144	General Laboratory Area	Misc. Horizontal Surface Area
1034T - Wipe	Top of furnace room 144	General Laboratory Area	Misc. Horizontal Surface Area
1062N - Wipe	Inside FE-9 on fan room 248	General Laboratory Area	Exhaust Ventilation System

Table 2.2 (continued)

Sample ID and type	Sample location	Building area	HA
1063R - Wipe	Inside FE-9 on fan room 248	General Laboratory Area	Exhaust Ventilation System
1064T - Wipe	Inside FE-9 on fan room 248	General Laboratory Area	Exhaust Ventilation System
1065W - Wipe	Inside FE-10 on fan room 248	General Laboratory Area	Exhaust Ventilation System
1068A - Wipe	Inside FE-10 on fan room 248	General Laboratory Area	Exhaust Ventilation System
1172Y - Wipe	Vent fin on new FE-1 room 136A	Beryllium Area	Exhaust Ventilation System
1173A - Wipe	Inside new FE-1 room 136A	Beryllium Area	Exhaust Ventilation System
1174C - Wipe	Vent fin on new FE-1 room 141	Beryllium Area	Exhaust Ventilation System
1186X - Wipe	Top of equipment room 136A	Beryllium Area	Misc. Horizontal Surface Area
1187Y - Wipe	Vent fin on new FE-1 room 141	Beryllium Area	Exhaust Ventilation System
1213R - Wipe	Vent fin on new FE-1	Beryllium Area	Exhaust Ventilation System

Table 2.3. Summary of quality control (duplicate) samples for wipes

Item	Room/elevation/ location	Sample no. / QC sample no.	Be (ug/t ²)	Li (ug/t ²)	Ni (ug/t ²)	Ti (ug/t ²)	U (ug/t ²)
Exhaust duct (inside)	141/7 ft S wall, center	1187Y 1188A	26.6 15.6	3.75 ND	11.9 30.7	1.24 ND	20.2 <62.8
Exhaust duct (inside)	248/3 ft at fanhouse	1066X 1067Y	17.7 16.8	13.1 11.3	119 93.2	4.22 2.82	2010 2100
Exhaust vent (inside)	141/7 ft S wall, center	1189C 1190F	1.35 10.7	ND ND	12.1 16.1	ND ND	63.5 <62.8
Supply vent (outside fin)	136A/18 ft/E side	1175F 1185W	224 178	5.78 2.89	5.56 3.20	ND ND	ND ND
Supply vent (inside)	130/9 ft /SW corner	1055F 1056G	0.20 ND	11.6 10.5	27.1 17.0	ND ND	<62.8 <62.8
Supply vent (inside)	137/7 ft /E wall, center	1088J 1089K	ND <0.14	ND ND	1500 1670	ND ND	<62.8 <62.8
Supply vent (inside)	148/8 ft /S wall, 2nd vent	1095W 1096X	ND ND	<2.76 <2.76	12.0 16.4	ND ND	ND ND
Supply vent (outside)	144/25 ft /N wall, 6th vent	1107N 1108R	ND ND	3.99 3.26	12.6 17.5	ND ND	<62.8 <62.8
Supply vent (inside)	113/8 ft /Center	1076M 1077N	0.15 0.24	6.89 7.62	15.0 29.3	ND ND	ND 91.0

Table 2.3 (continued)

Item sampled	Room/elevation/ location	Sample no. / QC sample no.	Sample concentration/QC sample concentration/relative difference				
			Be (ug/ft ³)	Li (ug/ft ³)	Ni (ug/ft ³)	Ti (ug/ft ³)	U (ug/ft ³)
Supply vent (inside)	116/8 ft /S wall, center	1083A	0.28	7.26	20.2	ND	<62.8
		1084C	0.62 76%	6.89 5%	17.6 14%	ND	ND
Wall	141/5 ft /S wall, center	1206G	<0.14	3.61	<1.08	ND	68.7
		1207H	ND	4.33	<1.08	ND	<62.8
Wall	144/2 ft /NE corner	1027H	ND	4.71	1.73	<3.48	89.3
		1028J	ND	4.71	2.64	6.06	<62.8
Base of rotating equipment	141/1 ft /S wall, center	1199T	19.8	ND	10.2	ND	66.0
		1200W	23.2 16%	ND	6.04 51%	ND	<62.8
Laboratory hood	141/7 ft /S wall, center	1197N	21.6	4.33	3.19	ND	<62.8
		1198R	17.2 23%	<2.76	3.44 8%	ND	ND
Vent grill (outside)	136/25 ft /First from W wall	1110W	<0.14	3.92	8.95	<3.48	<301
		1111X	ND	3.92	10.9	5.68	73.1
Light fixture	136/18 ft /Third from W wall	1131G	<0.14	21.0	39.0	<3.48	ND
		1132H	<0.14	21.7	45.7	ND	ND
Storage cabinet	136/7 ft /SW corner	1126X	0.54	8.91	60.9	ND	<62.8
		1127Y	0.32 52%	9.26 4%	57.4 6%	<3.48	103
Glove box	136/7 ft /NW corner	1123R	1.67	7.84	16.3	4.32	109
		1124T	0.51 106%	10.7 31%	13.8 16%	<3.48	82.0
							28%

Table 2.3 (continued)

Item sampled	Room/elevation/ location	Sample no. / QC sample no.	Sample concentration/QC sample concentration/relative difference				
			Be (ug/ft ²)	Li (ug/ft ²)	Ni (ug/ft ²)	Ti (ug/ft ²)	U (ug/ft ²)
File cabinet	138/5 ft /SE corner	1240L	<1.39	3.21	2.43	<3.48	88.7
		1241M	0.37	3.92	2.81	ND	ND
Light fixture	116/7 ft /S wall, middle	1232Y	12.1	26.0	46.0	<3.48	<62.8
		1233A	16.7	22.9	60.1	<3.48	<62.8
Bulk dust FE-9 (inside)	N duct		32%	13%	27%	---	---
		1142Y	3.61m	ND	878m	0.40m	NQ
		1144C	3.99m	ND	1060m	0.49m	NQ
			10%	---	19%	20%	---
Floor paint	144 Center	1008A	0.04m	2.28m	20.8m	ND	4.06m
		1010F	0.26m	2.04m	23.1m	ND	6.82m
			150%	11%	10%	---	51%

ND = not detected

NQ = not quantified

m = concentration is in mass units (ug/g)

A method blank is analyzed by the laboratory as part of their QC program. If an analyte is detected in a method blank at a concentration greater than the reporting limit, then the result is flagged with a "B." When reviewing the tables presented in Sect. 3, the "B" qualifier is sometimes noted. An example for one of the higher method blank results is for sample 1041G, where the lithium result is reported as 0.460 B $\mu\text{g}/\text{filter}$. In this example, the method blank had a reported concentration of 0.383 $\mu\text{g}/\text{filter}$ and the detection limit was 0.297 $\mu\text{g}/\text{filter}$. Because the LOC is significantly higher than that detected in the method blank, this error will not influence a decision based on the LOC. Based on a review of data with the method blank qualifier, contamination found in the method blank was never close enough to the LOC to impact a decision.

Information was documented in accordance with the S&T Plan. The field logbook that contains this information is included as Appendix A of this report.

2.2 PCB WIPE SAMPLES

PCB wipe samples were gathered in accordance with the S&T Plan. A total of seven oil stains were noted in the building. All stains were sampled for PCB analysis using a field immunoassay kit. Radian Procedure TP-307-9, *Immunoassay Screening Test for PCBs, PCPs, and PAHs*, was followed when collecting and analyzing these samples. No problems or discrepancies were encountered when obtaining these samples. QC samples were analyzed using gas chromatography analysis to verify immunoassay results. No QC exceptions were noted. The information recorded during field sampling activities is included in Appendix A.

2.3 BULK DUST SAMPLES

The collection of bulk dust samples provides significant information regarding potential concentrations of toxic elements in the dust at different locations in the building. All sampling was conducted in accordance with the S&T Plan, although in certain areas it was difficult to collect enough dust for analysis. A total of five samples were gathered from these areas.

Samples were gathered by collecting the material into a glass jar. No major problems or discrepancies were encountered during field sampling activities; however, several problems were encountered when analyzing the samples. The samples contained certain exotic metals that interfered with the ICP analysis. Some analyses were reported as negative concentrations, and others had concentrations reported over 100%. To overcome these problems, the samples were reanalyzed using ICP-MS. Samples with interference problems are included in Table 2.2.

QC samples were collected for one of the bulk dust samples. Results from the QC samples are all within 20% of the original sample for the standard suite. Therefore, the QC samples verify that bulk dust analyses were within the required confidence bounds.

2.4 ROOF SAMPLES

A total of five roof samples were collected for metals analysis, four for PCB analysis, and four for thorium analysis. No duplicate samples were submitted for metals analysis; however, duplicate samples were submitted for PCBs. PCBs were not detected in any of the analyses; therefore, PCBs are not a concern. The information recorded during field sampling activities for the roof samples is included in Appendix A.

2.5 PAINT SAMPLES

A total of eight different paint colors were identified in the building. Samples were collected for all paint colors plus one QC sample in accordance with the S&T Plan. Chisels and knives were used to scrape off the paint for collection. No major discrepancies or problems were encountered when collecting these samples. The results from the QC sample are shown in Table 2.3, except for lead and chromium. The relative difference among samples for the seven analytes was within 50%, except for beryllium and uranium, which had relative differences of 149 and 51%, respectively. Because these two metals are not associated with paint, they were probably encapsulated when the area was painted. A copy of the information recorded when these samples were collected is included in Appendix A.

2.6 PERCHLORATE RISK SURVEY

Perchlorate crystals sometimes form in exhaust ventilation systems that support laboratory hoods where perchloric acid was used. This is a shock-sensitive material that if not handled appropriately can cause physical injury during duct and lab hood dismantlement work. Therefore, it is very important to determine whether perchlorates could be present in the ductwork.

A one page questionnaire was developed to evaluate the likelihood that perchlorates might be present in either the hoods or ductwork. This questionnaire not only asks whether perchloric acid had been used, but how it may have been used and the washdown capabilities of the hoods. If no evidence of perchlorates is noted during the initial survey, sampling of the ductwork for other contaminants would proceed. Then, while collecting samples, the ductwork was visually examined for perchlorates to verify their absence. A copy of the questionnaire used is provided in Fig. 2.1.

**Industrial Hygiene Evaluation of Laboratory Hood for
Perchloric Acid**

BUILDING _____

ROOM NUMBER _____

DATE _____

BUILDING SUPERVISOR _____

HOOD EVALUATION:

1. Washdown capabilities: Yes _____ No _____
 2. Currently use Perchloric Acid: Yes _____ No _____
 - a. If so, how is Perchloric Acid used: Hot _____ Cold _____
 - b. Frequency and Comments: _____
 3. Used Perchloric Acid in past: Yes _____ No _____ Suspect _____
 - a. If so, how was Perchloric Acid used: Hot _____ Cold _____
 - b. Comments: _____
 4. Does hood have drains: Yes _____ No _____
Unknown _____ Process _____ Sanitary _____ Storm _____ "Hot" _____
 5. Visible crystals present: Yes _____ No _____
 6. Perchloric Acid sign present: Yes _____ No _____
 7. General Comments: _____
- SOURCE OF INFORMATION: _____

DUCT EVALUATION:

1. Perchloric Acid sign present: Yes _____ No _____
 2. Duct present: Yes _____ No _____
 3. Fan present: Yes _____ No _____
 4. _____
- Comments: _____

INVESTIGATOR: _____

Fig. 2.1. Perchloric acid questionnaire.

2.7 PHYSICAL HAZARDS ASSESSMENT

Potential physical hazards were identified in the building using the form developed for the S&T Plan. Hazards were identified for each room of the building. The completed forms for this assessment are included in Appendix B. No modifications, minor or major, from the S&T Plan were necessary to complete this survey.

2.8 HISTORICAL DATA

Two sources of historical data are evaluated in this report. The first source includes a radiological survey conducted by LANL personnel between August 19 and October 14, 1995, and the second includes quarterly wipe sampling activities conducted in the beryllium area between July 19 and October 30, 1995. The radiological survey was conducted through the analysis of smear samples taken from the floor, walls, and other surfaces. Results from these surveys are contained in Appendix C.